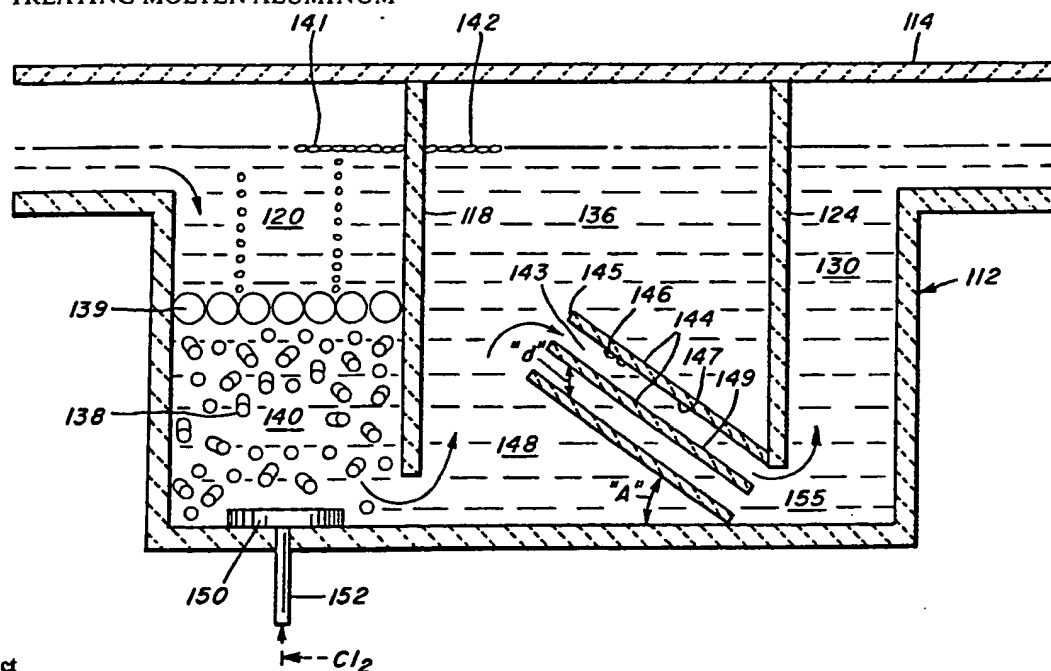


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(54) Title: TREATING MOLTEN ALUMINUM



(57) Abstract

Molten metal, such as aluminum^A is passed through a media of submerged contacting surfaces (138). Operating life is enhanced by periodic cleaning of the contact media (138) by a high flow gas purge. Molten metal such as the light metals aluminum and magnesium containing finely divided suspended particles may also be treated to remove particles. The molten metal containing such suspended particles is moved through the medium of submerged contacting surfaces (138). The molten metal preferably is then passed through inclined channels or passages in contact with inclined surfaces such as parallel plates (144). Buoyant particles and agglomerates collect on the underside of the inclined surfaces (147).

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TREATING MOLTEN ALUMINUM

This invention relates to a method of treating molten metal containing suspended particles for removal of said particles.

5 U.S. Patent 2,840,463 to Stroup et al describes a process where molten aluminum is filtered through a bed of refractory bodies to remove suspended solids from molten aluminum. U.S. Patent 3,039,864 to Hess et al describes a process wherein argon or other nonreactive gas is passed
10 through a bed of refractory bodies in countercurrent flow contact with molten aluminum to remove nonmetallic impurities and hydrogen gas from molten aluminum. That process was readily capable of removing high amounts of dissolved hydrogen gas, along with nonmetallic impurities, to very
15 substantially beneficiate molten aluminum. U.S. Patents 3,737,303, 3,737,304 and 3,737,305 to Blayden et al describe an improvement over the process of Patent 3,039,864 which provided for a very substantial increase in refractory body bed life along with other operating benefits and efficiencies and
20 has enjoyed considerable commercial success. According to that improved process, a small amount of chlorine or other chlorinaceous gas, along with larger amounts of nonreactive fluxing gas, is passed through the refractory media in contact with the molten aluminum. The extended life accord-
25 ing to the Blayden et al improvement typically eliminated the need to disrupt a casting operation in order to replace the filter media which could be done during interruption for another purpose such as adjusting or repairing a casting mold. However, as the useful life of molds and other cast-
30 ing associated equipment was increased over the years, it



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became apparent that still further increases in the useful life of the filter media for molten aluminum could be highly useful in still furthering the efficiencies and productivity in processing and casting molten aluminum and other 5 metals.

In addition, this invention relates to separating a molten metal such as aluminum or aluminum alloy from suspended fine particles typically comprising a buoyant phase such as a buoyant liquid salt phase and to the use of such 10 in combination with processes for purifying molten aluminum which produce a salt reaction product.

Molten metal such as aluminum, including alloys containing over 50% aluminum, has been treated with salt to remove impurities or with a gas which reacts with certain 15 impurities to produce a salt reaction product, typically liquid salt, or combinations of liquid salt with solids and gases. Processes of this type are described in U.S. Patents 3,767,382, 3,849,119 and 3,839,019. Each of these processes includes some provision for settling such as separation 20 chambers to separate treated molten aluminum from salt, either added in treating the molten aluminum or formed as a reaction product in treating the molten aluminum. However, as such systems are pressed for increased productivity, difficulties can be encountered in separating the suspended 25 salt-bearing and other particles from the molten aluminum. If the particles are not adequately separated and are carried by the molten aluminum to the casting station, such can result in surface and subsurface defects in the cast ingot.

The particles referred to are in the micron size 30 range, typically from less than 1 micron up to 40 or possibly occasionally 50 microns in size (1 micron = 10^{-4} cm). The particles can comprise liquid salt droplets or solid particles such as solid oxide particles or solid salt particles attached or encapsulated in liquid salt. The particles typically are 35 lighter or heavier than the molten metal and would be gravity separable but remain entrained or suspended largely because of the surface effects caused by their small size. Other



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than for their small size, the buoyant particles would rise to the surface for removal by skimming or similar operations and the heavier particles would settle downwardly out of the molten metal. However, efforts to date to remove the particles directly have not been entirely satisfactory. For instance, movement through a filter bed of the type shown in U.S. Patent 3,039,864 can result in premature plugging of the filter bed or, in some cases, passage of liquid particles to the casting operation. The salt particles typically result in oxide patches on continuously cast ingot which can give rise to problems in rolling the ingot into plate or sheet products.

In accordance with the invention, molten aluminum or other molten metal is moved through a media of submerged contacting surfaces such as a filter bed. The contacting surface media is selected so as to provide a high void fraction of one-half or more and a high specific surface area such as 50 sq. ft. per cubic ft. of media. A packed bed of Interloc saddles or Raschig rings provides a suitable medium. The molten aluminum or other metal moves through the contacting medium at a low velocity and a gas may be contacted with the molten metal moving through the medium. As the molten metal travels through the medium, entrained nonmetallic particles, such as oxide particles in the case of aluminum, are effectively removed provided the metal does not move through the media at too high a velocity. After a period of operation as described, the media may be periodically purged by passing therethrough a significant quantity of gas so as to disturb the bed and dislodge therefrom impurities causing them to rise and float upon the molten metal. This practice of particle removal within the media and periodic purging and disturbance of the media to flush trapped particles therefrom has enabled the improved process to demonstrate markedly improved operating life even over that of the highly successful Blayden et al process as described in U.S. Patent 3,737,305.

Further in accordance with the invention, molten



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aluminum or other metal containing suspended finely divided particles typically comprising buoyant liquid such as liquid salt or comprising other salt or suspended phases can be treated to coalesce or agglomerate the suspended particles
5 so that they are more readily separated by gravity from the molten metal. Where the molten metal is aluminum, an agglomerated particle size of over 50 microns, preferably over 60 microns, typically facilitates separating by gravity even where the aluminum is moving provided it is relatively quietly. Where
10 the agglomerated particles are buoyant, their buoyancy causes them to rise to the surface for convenient separation by skimming or the like. Where the agglomerated particles tend to sink, they can be retained in a trap or other provision. The molten aluminum or other metal is moved through a
15 medium of submerged contacting surfaces such as a bed of packing, suitably refractory packing such as alumina packing, having a high void fraction and a high specific surface area such as Interloc saddles or Raschig rings. The molten metal moves through the contacting medium at a relatively
20 low velocity, and a gas may be passed upwardly through the medium concurrently or countercurrently with the metal. Coalescence or agglomeration of entrained fine particles occurs within the medium, and the coalesced larger particles are separated from the molten metal by gravity, for instance
25 rising to the surface because of their buoyancy.

Further coalescence and agglomeration of particles or collection thereof may be made to occur by moving the molten metal preferably downwardly and laterally in contact with inclined surfaces, suitably substantially parallel
30 surfaces. Buoyant particles collect on the inclined surfaces and typically travel laterally and upwardly in counterflow relationship to laterally and downwardly flowing molten metal. Coalesced buoyant salt-bearing phase is thus removed from the zone of the inclined surfaces by its buoyancy and
35 rises to the surface of the molten metal for removal by skimming or the like. Heavy agglomerated particles sink and can be removed by a suitable trap or other arrangement.



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In this description reference is made to the drawings in which:

Figure 1 is a schematic elevation view in cross section illustrating the present invention.

5 Figure 2 is an elevation view in cross section schematically depicting the operation of an embodiment of the improved system and schematically showing one arrangement suitable in practicing the invention.

Figure 3 is a highly schematic elevation illustrating an embodiment of the invention in association with the process shown in U.S. Patent 3,839,019.

Figure 4 is a highly schematic elevation illustrating an embodiment of the invention.

Referring to Figure 1, molten metal enters the treatment vessel 10 through inlet 12 and passes downwardly in the down leg 14 on the inlet side of the baffle 16 which divides the vessel 10 into down leg 14 and up leg 18. In the vessel 10 is situated a zone of noncontaminating contact surface media such as a packed bed. The molten metal passes downwardly through the submerged contacting media 20 in down leg 14, passes beneath baffle 16 and then moves upwardly through up leg 18 and exits through outlet 21. As the molten metal passes through the media 20 it may be contacted with a stream of gas which enters through disperser 22. In the embodiment shown in Figure 1 gas entering through disperser 22 rises upwardly within down leg 14 and the media zone 20 therewithin in countercurrent flow relationship with the molten metal moving downwardly through down leg 14. Downward movement of the molten metal through the contact media zone 20 is preferred, although upward movement can be utilized.

In the case of treating molten aluminum, gas introduced through disperser 22 can comprise a nonreactive gas or a halogenaceous or chlorinaceous gas or mixtures thereof. For aluminum, the nonreactive gas can be any of those disclosed in the Hess et al patent including the inert gases of the periodic table, helium, neon, argon,



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krypton and xenon and mixtures thereof, with argon being preferred because of its cost and availability. In addition, nitrogen or carbon dioxide may be employed, although precautions are often warranted to avoid the formation of

5 nitrides, oxides, carbides or complexes thereof. All these gases are considered nonreactive in the practice of this invention for treating molten aluminum. Halogenaceous gases such as freons can be employed as well as chlorinaceous gases such as chlorine, aluminum chloride and hexachloro-

10 ethane, although chlorine is a somewhat preferred chlorinaceous gas because of its cost and compatibility with existing facilities in many existing installations. A typical gas mixture could comprise major portions of argon and minor portions of chlorinaceous or halogenaceous gas such as 1

15 to 50, typically 1 to 10, parts of chlorinaceous or halogenaceous gas and about 99 to 50, typically 99 to 90, parts of a nonreactive fluxing gas on a volume basis. However, other mixtures can be useful, such as mixtures

approaching or even exceeding equal portions of chlorinaceous

20 or halogenaceous gas with nonreactive gas. It is desired that any gas mixture be premixed prior to entering zone 20 as indicated in the Figure which shows the gases being mixed before passing through disperser 22.

The amount of fluxing gas for treating molten

25 aluminum varies from about 0.005 to about 0.5 standard cubic foot per hour (S.C.F.H.) per square inch of cross-sectional area in zone 20 in a plane normal to the gas travel, that is, the horizontal plane in the Figure which is normal to the upward gas flow and to the downward overall

30 metal flow. Preferred gas flow rates are 0.015 to 0.2 SCFH per square inch. The aforementioned gas flow rates are those which apply while treating molten aluminum in accordance with the invention. As will be explained hereinbelow, a larger gas rate is employed to periodically purge

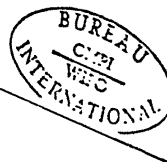
35 zone 20. It is desired that disperser 22 occupy a substantial portion of cross section beneath the contact media zone 20 so as to provide for a wide uniform dispersion of the gases



through the main contact zone 20. Thus either a large disperser 22, as depicted in Figure 1, can be employed or a plurality of smaller dispersers. The use of wide zone gas dispersion can make it advisable to employ inclined baffles 17 beneath major baffle 16 which channels metal flow under baffle 16 in a generally downwardly-inclined fashion which reduces the amount of gas which can pass beneath baffle 16, thus tending to retain gas within main contact zone 20 where it can more advantageously provide laterally contact zone 20. Thus, it is preferred to conduct molten metal downwardly inclined flow means to reduce the amount of gas from the gas contact zone, which in the Figure 1 embodiment is the main contact zone 20 thus serving to conduct liquid metal flow but restricted gas flow from zone 20.

In accordance with the invention, it is important to properly select the contacting surface media 30 for main contact zone 20. A first requisite for this submerged contacting surface media is that such have a relatively high void fraction, meaning fraction of total volume which is not occupied by solid material such as the packing or submerged bodies and hence available for molten metal movement through the contact surface according to the invention should be about 0.4 or about one-half, suitably about 0.6. A preferred void fraction is about 0.7 or 0.8 or more. A void fraction of 0.6 is almost twice that of a filter bed made up of 3/4-inch diameter alumina balls or a filter (U.S. Patents 3,737,305 and 3,039,864), each of whose void fraction is about 0.33. The high void fraction in accordance with the invention facilitates attachment of fine nonmetallic particles and other particles to the contact surfaces for removal thereof from the molten metal moving slowly through said contact zone 20.

A second requisite for the contact media 30 is



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that it have a high specific surface area (area per unit volume) which provides surfaces for the desired nonmetallic particle removal. In accordance with th invention, the surface area desired for the contact media is a minimum
5 specific surface area of at least 25 sq. ft. per cu. ft., with a specific area of 50 or 75 sq. ft. per cu. ft. being more suitable and with specific areas over 80 being preferred. Specific contact media areas of over 90 sq. ft. per cu. ft. appear to provide superior results. Provided such can be
10 accompanied by adequate void fraction, a specific area of 120 sq. ft. per cu. ft. is more preferred. The following Table 1 sets forth suitable packing materials (Interloc saddles and Raschig rings) in accordance with the invention, along with comparison materials with respect to their
15 respective void fraction and average specific surface area. The comparison materials are those set forth in U.S. Patents 3,737,305 and 3,039,864.



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Table 1

<u>5 Packing</u>	<u>Average Bed Void Fraction</u>	<u>Average Specific Surface Area (ft²/ft³)</u>
1/2" Interloc. saddles	0.78	190
1/2" x 1/2" Raschig rings	0.85	93
3/4" diameter balls	0.33	54
10 -6+14 mesh particles	0.33	257

It can be seen in the foregoing Table 1 that 3/4-inch diameter balls or fine mesh particles such as those depicted in the aforesaid patents are not suited in practicing the invention. Beds made of these materials can eventually become clogged so as to cause the surface of molten metal in the inlet side 12 to rise above that shown in outlet zone 21 which is caused by the pressure drop through zone 20. In such prior practices, once the level on the inlet side 12 starts to rise excessively higher than that in the outlet zone 21, such was irreversible and steadily increased to eventually cause interruption of the operation because of inadequate molten metal travel through the treatment zone. The more open type bed provided by saddles or rings, however, serves the purpose of the invention. Rings may be provided by cutting pipe-like or hollow cylindrical shapes into relatively short segments.

The material selected for contact media 30, such as the Raschig rings or Interloc saddles, should not contaminate the molten metal and have a long surface life in exposure to molten metal without melting or deteriorating so as to interfere with the improved process or desired results. Where the molten metal is aluminum, typical temperatures are 1250° to 1500°F., and the media 30 should be able to withstand such. Suitable refractory materials for use with aluminum having a higher melting point than aluminum and being substantially inert toward aluminum include such substances as chromite, corundum, forsterite, magnesia spinel, periclase, silicon carbide and zircon. Alumina



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(synthetic corundum) is a preferred noncontaminating material for molten aluminum. Carbonaceous materials such as fashioned from used carbon anodes can also be useful with molten aluminum, although such tend to float, and some provision such as refractory screen may be provided above zone 40 to prevent the carbonaceous material from floating out of the zone. Hence, the term "noncontaminating" is intended to include both refractory materials and even carbonaceous or other materials which may not be considered completely refractory to aluminum in the strict sense of the term "refractory" but are sufficiently stable that they do not introduce unwanted contaminants into the molten metal.

The depth of contacting media 30 is at least six inches and preferably 10 or 15 inches or more. A bed of about 20 inches is desirable. This provides desired time for contact between the molten metal and the contact media surfaces to encourage removal of nonmetallic particles and to allow for sufficient time for contact between the metal and any gases introduced into contact zone 20.

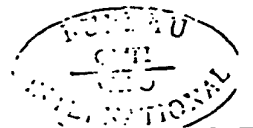
As the metal moves through contact zone 20, it is desired that the metal move at a relatively low velocity. The superficial molten metal velocity (velocity based on assuming no media or packing 30) through zone 20 is suitably less than one-half ft./minute. A slower superficial velocity of less than 0.4 or 0.3 ft./minute is preferred, for instance a superficial velocity of about one-fourth ft./minute is satisfactory. However, on a somewhat less preferred basis, molten metal velocity of up to three-fourths or one ft./minute can provide for useful results. However, for the particular arrangement depicted in Figure 1 which shows no further significant provision for particle removal after exiting zone 20, a metal velocity of not over one-half ft./minute is considered better. Particles coalesce and are trapped in the media thus removing them from the treated molten metal, and this combined coalescence and removal effect is enhanced by relatively slow flow rates.

As indicated hereinabove, the practice of the



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invention includes introducing fluxing gases, including fluxing gas mixtures, into contact zone 20 for treatment of molten aluminum. Where the gas mixture includes a halogen-aceous or chlorinaceous gas, such can remove trace impurity elements such as sodium and calcium as well as assist in removing oxide and dissolved gas impurities. Such gas treatments usually involve relatively slow rates such as around 0.05 SCFH per square inch of bed cross section normal to the plane of overall metal and gas movement through the bed (i.e. measured in the horizontal plane). However, the invention also includes the periodic use of gas rates two or three or more times this order so as to disturb the media 30 and purge or dislodge therefrom particles trapped or contained therein previously removed from the molten metal so as to cause said particles to rise and collect as a floating layer 36. Suitable purge gas flow rates are about 0.008 or 0.009 to about 0.6 or 0.7 or more SCFH per square inch, suitable about 0.025 to 0.35 SCFH per square inch, and adequate to disturb the media and dislodge particles therefrom. Because of the serious disturbance of the media 30 caused by such copious gas flow, it is preferred to provide some sort of overlying heavy material such as a single layer of three-quarter or one inch refractory balls 34. This prevents the relatively high gas flow rates used to purge zone 20 from forcing contact media members, such as Raschig rings or Interloc saddles 30, from being carried out of zone 20 and possibly settling back downwardly in a non-uniform and nonpacked array. Hence, the invention includes the practice of periodically purging the bed by use of a gas flow which disturbs media 30 to dislodge and remove particles therefrom. This purging can be performed at any point where convenient. For instance, it can be deferred until the molten metal level on inlet side 12 is a significant amount higher than that in exit zone 21. However, it is not necessary to wait to this point. The purging can be done at any convenient point such as during any interruption in metal flow such as during any interruption in casting or any ant c dent or subsequent operation which causes a delay or



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interruption in molten metal movement through the improved treatment vessel 10. It is preferred that during the purging operation molten metal movement through zone 20 be interrupted such that it then becomes most convenient to perform the purge during an interruption in molten metal travel caused by antecedent or subsequent operations. However, if the associated casting operation is completely continuous and not amendable to any interruption, the ingot cast from the metal passing through zone 20 during purging might contain impurities which lower its quality. It is to be understood that it is not practical to purge a bed such as that shown in U.S. Patent 3,737,305 and utilizing a bed of fine mesh refractory bodies such as 3 to 14 mesh size since the high gas flow rates are incompatible with the relatively small void fraction of such filter beds and is highly disruptive thereto. That is, the process in accordance with Patent 3,737,305 involves some continuous flushing of impurities from the fine particle size filter bed. However, this continuous flushing, while effective to provide for increased bed life in that system, still allows for some accumulation of nonmetallic particles within the filter bed which eventually causes the same to exhibit increasing pressure drop and increasing buildup of molten metal head from inlet side 12 across baffle 16 to outlet side 21 whereby the level in inlet side 12 can rise several inches above outlet level 21. However, once this metal head differential starts to occur in the process according to Patent 3,737,305 it is normally irreversible and leads to eventual bed replacement. The present improvement in contrast can be repeatedly purged by the high gas rate purge practice and exhibit still further and even markedly extended bed life approaching indefinite bed life in some applications. In practicing the invention extended runs with no buildup of metal head from level 12 to level 21 have been observed.

Referring now to Figure 2, the improved system 110 includes a containment vessel 112 constructed or lined with a suitable refractory or a nonreactive material and provided



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with a lid 114. Inlet baffle 118 establishes inlet leg 120 and outlet baffle 124 establish s outlet leg 130. Within the inlet leg 120 is a zone 140 of submerged contacting surfaces, such as packing, as shown, having a high void fraction and a high specific surface area. Packing 138, like Interloc saddles or Raschig rings, provides suitable contacting surfaces and can be held in position by larger ball members 139. Alternatively, the contacting surfaces can be positioned in zone 148; that is, to the right of baffle 118 as shown in Figure 2, or packed beds can be positioned on both sides of baffle 118. If the bed is positioned only in zone 148, baffle 118 should be moved to the left in Figure 2. The submerged contacting surfaces or packed bed provides a first treatment zone 140 according to the embodiment shown in Figure 2 and may include a gas disperser 150 within or beneath the packing 138 to introduce a gas or gas mixture provided through gas supply line 152. Where packing 138 occupies zone 148 in lieu of or in addition to the bed in leg 120, a gas disperser may be provided for that bed if desired.

A second treatment zone 136 is shown in Figure 2 between the inlet and outlet baffles 118 and 124. Situated within zone 136 are parallel plates 144 which are inclined downwardly and toward the outlet leg 130. While three plates 144 are shown in Figure 2, there may be a greater or lesser number as desired or required according to the guidelines herein set forth.

In operation, molten metal containing fine agglomerable particles, typically less than one micron up to 40 or possibly 50 microns, travels down inlet leg 120 and through first zone 140 containing submerged surface contact media such as packing 138. Within zone 140 there occurs a substantial amount of coalescence or agglomeration of the suspended salt or other fine particles into larger particles typically over 50 or 60 microns. Where the suspended fine particles are buoyant, they agglomerate into larger particles within zone 140, many of which particles rise to the surface as a floating phase 141. Some agglomerated buoyant particles



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may pass under baffle 118 and rise within chamber 136 as a floating phase 142. Where the particles are heavy or tend to sink, they tend to collect beneath zone 148 and can be periodically removed. After passing through the zone 140, preferably the molten metal is moved downwardly and toward the outlet leg 130 passing through channels 143 between parallel plates 144. The metal moves relatively slowly between the plates 144 under substantially laminar flow conditions. As the molten metal travels between the plates 144 in laminar flow fashion, buoyant particles, including particles coalesced in zone 140, collect and coalesce or agglomerate further to form agglomerated phase 146 on the underside surface 147 of the plates 144. The buoyant agglomerated phase 146 moves upwardly and laterally (to the left in Figure 2) in a direction counterflow to the metal flowing downwardly and laterally between the plates 144. The agglomerated buoyant phase 146 thus passes upwardly along the underside of plates 144 and upwardly off the upper edge 145 of the plates 144 to rise to the surface in the treatment zone 136 and accumulates as floating material 142 in zone 136. Where the particles sink, they collect at site 155 for periodic removal. Thus, the channels 143 between plates 144 provide for collection and further agglomeration of particles, typically agglomerated in zone 140, so as to further improve particle separation from the molten metal.

As shown in Figure 2, a gas can be introduced into the lower regions of zone 140 and contact media 138 through a gas dispenser 150 connected to a gas inlet pipe 152. Where the molten metal is aluminum, suitable gases may be relatively inert gases or nonreactive gases including the so-called inert gases such as helium, neon, argon, krypton and xenon or, on a less preferred basis, carbon monoxide, nitrogen, carbon dioxide or various other gases provided care is taken not to introduce contaminants into the molten aluminum. Argon is a preferred gas for aluminum because of its ready availability and ease of handling. If desired, the inert or nonreactive gas can be combined with a chlorinaceous gas or other halogen containing gas such as



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halocarbons (e.g. freons) in ratios of about 50 to 99% non-reactive gas and 50 to 1% chlorine or other chlorinaceous or halogenaceous gas. For instance, a mixture of 85 to 99% argon and 15 to 1% chlorine or freon is useful. The use of a chlorinaceous or halogenaceous gas can remove trace impurity elements such as sodium and calcium in the bed 140 as well as oxide and dissolved gas impurities. The gas can also serve to flush the media 140 and facilitate release of agglomerated buoyant particles therefrom to rise toward overlying layer 141. Periodically purging the media by increased amounts of gas flow can release agglomerated particles which may be trapped in the media, and one preferred practice of the invention includes use of a fluxing gas substantially continuously during operation and periodically increasing the gas flow rate to 1-1/2 or 2 to 3 times the normal continuous rate so as to disturb the bed sufficiently to purge trapped agglomerated particles from the media so as to rise to surface phase 141. During this high gas flush rate it is preferred that metal flow through zone 140 be interrupted because the disturbance of the media can interfere with proper cleaning of the molten metal. This is normally easily facilitated by scheduling media purges during down time such as for casting mold replacement or adjustment. By interrupting metal flow through zone 140 is meant that the overall flow is interrupted but allowing for some minor metal movement within zone 140 such as in response to the high flow gas purge.

In zone 140 it is desired to move the molten metal over large surface areas at a relatively slow velocity so as to facilitate the desired agglomeration of salt and other fine particles. The superficial molten metal velocity (velocity based on no media or packing 138) through this zone 140 is suitably less than 5 feet per minute. A slower superficial velocity of less than 3 feet per minute is better and less than 1 or 2 feet per minute is preferred. For molten aluminum, a velocity of one foot per minute corresponds to 60 pounds per hour per square inch of cross section in zone 140 measured in a horizontal plane (normal to mean metal



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path). Molten metal retention time in zone 140 should be 1/4 or 1/2 minute or more with times of 3/4 minute or more being preferred.

One preferred practice is to employ sufficient metal velocity through the contact media to flush agglomerated particles from zone 140 with molten metal movement. A superficial velocity of about 1/2 foot per minute or more, for instance 0.55 or 0.6 or 0.65 foot per minute or more, is preferred. This can hinder agglomerated particles from accumulating in zone 140 to the extent that molten metal movement is resisted. This preferred embodiment results in the molten metal exiting the contacting surface zone carrying with it substantial amounts (or even major portions or the majority, for instance 60% or more) of particles agglomerated in zone 140. However, the fact that the larger agglomerated particles resulting from the improvement are separable by gravity, even from moving molten metal, enables ready removal of the particles and, hence, such molten metal desirably is passed through a gravity separation zone after exiting the contact media. Such a zone is preferably provided by inclined plates 144 which are highly efficient to remove agglomerated particles carried out of the contact surface media by the molten metal. While some of the particles, e.g. oxides, have a chemical makeup suggesting a density above that of the molten metal, such as aluminum, the open porous structure of the agglomerated particles often gives them a low bulk density which causes gravity separation by upward (or buoyant) movement of the particles from the metal. This facilitates ready removal from floating phase 142 wherein oxides, salts and other phases are kept floating on top of the molten metal by the surface tension (interfacial tension) of the molten metal thus effectively trapping such for ready removal. This preferred practice greatly extends the life of the surface contact media. Further, it can combine with the preferred practice of periodically purging or flush disturbing the media by high gas flow rates to virtually eliminate need for contact media



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r placement. This advantage is available because of the high void fraction and specific surface area for the media in accordance with the invention. As an illustration of the improvement in useful contact media life, the system according to U.S. Patent 3,737,305 was situated after the system according to U.S. Patent 3,839,019 and was successful in enhancing the molten aluminum leaving the system of U.S. 3,839,019 and exhibited good filter bed life. Replacing the system of U.S. 3,737,305 with the present improvement using Raschig rings resulted in running five times longer and, capability-wise, even more since the operation was interrupted for reasons having no relation to bed life.

The inclined plates 144 shown in Figure 2 may be provided in any suitable number and in various configurations suitable in practicing the invention. The plates should be fashioned from a material which is not attacked by nor introduces contaminants into the molten metal. For treating molten aluminum suitable plate materials are graphite, silicon carbide, carbon, alumina or other materials which don't contaminate molten aluminum. The plates are shown as flat, but they can be corrugated in either direction, especially the direction normal to the plane of Figure 2. Also, instead of plates, the inclined surfaces may be provided by simply stacking an array of pipes in the general configuration shown in Figure 2. This would provide flow paths within the inside diameter of a round pipe and in the spaces between adjacent pipes (diamond-shaped or triangular-shaped depending on the stacking relationship of the pipes). Pipes could be of square, rectangular or polygonal section. The number of plates, or the number of spaces or channels between the plates, is a function of the desired overall flow rate through the system while providing a laminar flow condition between the opposed surfaces 147 and 149. The flow should be laminar without substantial turbulence or flow separation from the surfaces 147 and 149. The Reynolds number should not exceed 3500 and preferably is not in excess of 2800. Flow rates of 3 or 4 feet/minute or less



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through the channels between the plate members 144 are suitable with flow rates of less than 2 feet/minute being preferred.

The distance between the plates or between opposed surfaces 147 and 149 can vary somewhat in the practice of the invention. The plates should not be so close as to be readily clogged by the agglomerated materials nor should the spaces between plates present such a small flow path as to impede the flow of molten metal therethrough. As just stated, the metal flow rate moving through the spaces between the plates 144 should be quiet and laminar. A distance between opposed surfaces 147 and 149 of about 1/2 or 1 to 2 inches is suitable in practicing the invention, although distances of up to 10 inches or even more can be useful.

The angle A for the plates can also vary in accordance with the invention. The plates preferably should not be horizontal since such would impede the desired separating action which occurs between the plates (separating upwardly or downwardly depending on weight relative to the molten metal) and would impede the desired removal of agglomerated material from between the plates. In a broad sense, the invention includes angles of about 5 to 90° relative to the horizontal. However, an angle of 90° has some disadvantage as reducing the amount of particle removal and the angle is suitably less than 90°. An angle of about 10 or 15° to 70 or 80° is more suitable with angles of about 25 to 50° being preferred. Angle A of 20 or 30° to 45° is very suitable in practicing the invention, especially with a spacing of about 1/2 or 1 to 2 inches between plates or inclined surfaces in treating molten aluminum.

The length of the channels in the direction of metal flow should be such as to provide sufficient time to achieve the desired separation and agglomeration of particles to facilitate floating of the lighter particles and sinking of those heavier than the molten metal. Preferably, the ratio of the channel length to the vertical distance "d" between plates is at least 5:1, preferably 8 or 10:1, although a



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smaller ratio can also be useful in certain applications. Plates 144 may be substituted with hollow pipes or tubes or any other convenient method for providing inclined surfaces 147 and 149 defining a channel or flow path for downward and lateral movement of the molten metal in contact with such surfaces which serve as surface sites for the desired collection and agglomeration or coalescence of the suspended particles. While generally parallel plate or pipe surfaces are convenient for such purpose, the invention is not necessarily intended to be limited to this specific approach. What is important is that a downward, preferably laterally downward, flow path or channel be provided for molten metal to travel in contact with a surface at least partially defining such flow path. Preferably, upper and lower inclined surfaces define the upper and lower boundaries of an inclined molten metal flow path or channel. These surfaces are advantageously straight and smooth for simplicity of construction and to more readily facilitate laminar flow conditions and prevention of clogging. However, the surfaces 147 and 149 may be curved, corrugated, roughened or otherwise depart from the straightforward arrangement of Figure 2. Further, while substantially parallel surfaces 147 and 149 are preferred, such is intended in the broad sense. For instance, roughened or corrugated surfaces are taken to roughly describe a single or "average" plane and some amount of divergence or convergence such as not substantially greater than 10° or 15° is herein taken to be substantially parallel.

The improved process is highly suited to the treatment of molten aluminum and has demonstrated a marked ability to remove finely divided particles, especially finely divided particles comprising salt phases, particularly salt phases which are liquid or which are less dense than aluminum. While such salt phases tend to float in molten aluminum if possessed of sufficient size, they remain entrained in the aluminum where the particles are of the micron size and hence their agglomeration or coalescence in accordance with the improvement facilitates ready removal since the buoyant particles ultimately gather on the surface of the



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metal as a floating salt phase easily removed. The improvement is especially applicable to alloys of aluminum containing substantial amounts of magnesium, which alloys have been subjected to chloridizing or halogenizing treatments directed to the removal of trace elements such as sodium and calcium such as according to one embodiment of the process described in Patent 3,839,019, incorporated herein by reference. According to one embodiment of that process, molten aluminum or alloys thereof are contacted with a gas comprising a chlorinaceous gas such as a mixture of argon and chlorine (90% Ar - 10% Cl₂) to degas the aluminum, remove trace elements such as sodium and calcium and remove oxide impurity particles. However, where the aluminum alloy has a substantial magnesium content such as an Mg content above 2 or 3%, for instance an alloy containing 3-1/2 to 6% Mg, some small amount of liquid MgCl₂ is included with the otherwise solid CaCl₂ and NaCl formed by chloridizing the trace elements Na and Ca. The salt particles and other particles, because of the liquid MgCl₂, generally exhibit a liquid or semi-liquid character and would be buoyant if coalesced into larger sizes. However, as the treated metal exits the process according to Patent 3,839,019, some of these particles sometimes can remain entrained especially where the process is pressed to increase productivity by processing increased amounts of aluminum per hour and where more and more aluminum derives from contaminated scrap. In treating alloys of aluminum containing Mg, especially alloys containing 4% or more Mg, such dispersed phase can present problems in separation which can carry through into the eventual cast product. Situating a unit of the type depicted in Patent 3,737,305, after the process according to Patent 3,839,019 improves the overall operation quite substantially but sometimes the micron sized particles can shorten the bed life of the filter bed shown in Patent 3,737,305 and interfere with the bed life improvement otherwise associated with that process.

Hence, one especially useful embodiment of the improvement arises in combination with a process of the type



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in Patent 3,839,019 wherein molten aluminum, especially molten aluminum containing magnesium, is purified by chloridizing within a chloridization chamber so as to react chlorine or chlorides or other halides with metallic impurities, typically Na and Ca or even Mg, in the aluminum and produce the chloride or halide salts of those impurities, and wherein the chloridization is effected within an agitated region such as a region agitated by a rotating device such as a turbine blade about 12 inches in outside diameter with 2-inch square paddles and rotating at about 150 to 400 rpm. Such processes are useful in treating molten aluminum with a gas comprising a chlorinaceous gas such as a gas comprising about 90% or more argon or other nonreactive gas and 10% or a little less of chlorine. The improved process can produce significant improvements in the overall quality of the metal exiting such a treatment, and one suitable arrangement for combining the improvement with said process is depicted in Figure 3 which is a highly schematic "stick" diagram.

In Figure 3 molten aluminum is treated in chamber 212 by introducing a mixture of argon and chlorine and passing it down within a hollow conduit (not shown) provided within rotating shaft 252 and including at the lower end thereof a rotating blade 254. Chloridization in this fashion will react sodium, calcium, strontium, lithium and magnesium to form their respective chloride salts which, in large part, collect in overlying salt layer 210. For increased treatment, two or more such chloridization zones may be situated in parallel or series flow relationship. After exiting the chloridization chamber 212, the molten aluminum passes under baffle 223 and into a salt separation chamber 224 where it undergoes a direction change by the action of baffle 232. The metal then is moved downwardly through submerged contacting surfaces such as bed 220 in accordance with the first treatment according to the invention wherein the molten aluminum is contacted with a gas comprising argon and chlorine introduced through disperser 250. The bed 220 includes packing in accordance with the hereinabove set forth guidelines. The



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molten aluminum passes beneath baffle 218 and move upwardly through zone 248 which may likewise be provided with packing or other submerged contacting surfaces in accordance with the invention and the molten metal can be contacted with gas introduced through disperser 251. Fine salt particles passing downwardly through filter bed 220 are agglomerated into larger particles, some of which rise upwardly from the bed to join the overlying salt layer 210. This is enhanced by the fact that the particles exiting separation zone 224 entrained in the molten aluminum are buoyant except for their extremely fine size such that when agglomerated they tend to rise upwardly into overlying salt layer 210. This effect can be enhanced some by action of gas, introduced through disperser 250, as the gas moves upwardly through the bed 220. The molten aluminum passing beneath baffle 218 may be treated further by including a packed bed within zone 248 or simply allowed to rise upwardly to enter the upper extremities of the paths between inclined plates 244. If no packing is present in 248, some agglomerated particles passing under baffle 218 will simply rise upwardly into salt layer 242. If a bed is present in zone 248, further agglomeration will occur and the buoyancy of the agglomerated particles will carry some particles newly agglomerated as well as some agglomerated particles exiting bed 220 upwardly into the overlying salt layer 242. Next the molten aluminum is passed downwardly and laterally through the spaces between plates 244 wherein further agglomeration of the salt and other particles and collection of agglomerated particles leaving bed 220 occur. As already made clear, the flow through the spaces between plates 244 should be relatively slow and laminar. The molten aluminum is then passed upwardly through exit chamber 230 and out to the next operation, typically a casting operation.

The present improvement is also useful in combination with processes wherein aluminum is treated with salts with or without simultaneous gas treatment for oxide, gas or impurity metal removal, particularly where such a process



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involves vigorous agitation such as in Patents 3,767,382 and 3,849,119. Hence, the improvement is considered highly useful in combination with processes for treating molten aluminum with halogen or chlorine active agents wherein salt
5 reaction products are formed which are advantageously separated from the aluminum prior to casting.

While the downwardly inclined plates 144 in Figure 2 are often preferred, other arrangements can also be employed. For instance, referring to Figure 4, which is a
10 highly schematic "stick" diagram, the plates 344 are shown inclined upwardly in the direction of molten metal travel. Thus, molten metal enters the system and passes downwardly through bed 340 where it may contact a gas, not shown. After passing beneath baffle 318, the metal moves upwardly and
15 toward the right in Figure 4 between parallel plates 344. It then passes over baffle 325 and under baffle 324 and exits the system. Particles are separated between the plate 344 in a manner similar to that with the Figure 2 arrangement except that buoyant particles are moved con-
20 current with the metal flow between plates 344.



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Example 1

In any comparison it is, of course, advisable to use the same type of metal and metal quality (contamination or freedom from contamination) and the same flow rates and other operating conditions to provide a meaningful comparison. Such a comparison is readily apparent in the following example. In an arrangement as depicted in Figure 1, the process as shown in Patent 3,737,305 was used to purify molten aluminum. The filter bed included a portion of fine mesh (-6+14) alumina granules 13 inches deep situated upon a substrate of 3/4-inch alumina balls 6 inches deep. Molten aluminum was moved through the filter bed at a superficial velocity of about 0.2 ft./minute and contacted with a mixture of 3 parts chlorine and 100 parts argon at a gas flow rate of about 0.05 SCFH per square inch of bed cross section in the horizontal plane. The molten aluminum alloy was alloy 5182 containing 4 to 5% magnesium and 0.2 to 0.5% manganese, said alloy being widely used in tear open beverage can ends and readily available as scrap containing substantial amounts of impurities. The practice in accordance with Patent 3,737,305 was found to markedly improve the quality of the aluminum passing therethrough to render it suitable for casting into ingot for rolling into sheet suitable for can end use. However, as the process was used a gradual buildup in molten metal head across baffle 16 was observed and the process was interrupted after 160 hours because of head buildup.

The fine mesh particles and alumina balls were removed from the vessel and replaced with 1/2-inch Raschig rings made of alumina and having situated thereupon one layer of 3/4-inch alumina balls as shown in Figure 1. The same type 5182 molten aluminum metal of high contamination was run through this unit practicing the present invention which provided the same superior metal purification as achieved with the process of Patent 3,737,305 such that the metal exiting through exit 21 exhibited markedly reduced amounts of gas, nonmetallic impurity and trace element

100-100
C-10
100-100

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content. However, in practicing the improvement with the ring contact media, there was no buildup observed even after an extended run of 750 hours whereupon the process was interrupted for reasons having nothing to do with the process. During this run the high gas rate periodic purge was employed at a gas rate of 0.2 SCFH per square inch of bed cross section in the horizontal plane which amounts to about four times that used for the normal metal treatment. In each instance the periodic purge was employed during a period of interruption in metal flow because of casting interruption. No other maintenance or adjustment to the molten metal treatment process was necessary during this period and the molten metal flow rate, quality and all characteristics were the same after 750 hours as during the first hour of operation which verifies the marked improvement in operability of the present improvement.

Example 2

Thin sheet of aluminum alloy 5182 containing 4 to 5% magnesium, along with 0.2 to 0.5% Mn, is widely used in tear-open beverage can ends. It is produced by casting a relatively large ingot which is homogenized and hot rolled into thick sheet followed by cold rolling into thin sheet. When alloy 5182 is treated prior to casting by the process of Patent 3,839,019 to remove sodium and calcium, some of the magnesium present is converted to $MgCl_2$ in the form of very minute buoyant liquid particles. These particles can present some difficulty in removal. If substantial amounts of such particles are carried with the metal to the casting operation, the resulting cast ingot can exhibit surface patches of oxides of magnesium and aluminum. These oxide patches result in edge cracking during the subsequent rolling operations which can be so excessive as to impede rolling or even to render the ingot unacceptable. The presence of such oxide patches in serious amounts can prevent rolling as requiring removal of so much edge metal as to be uneconomical.

In a series of runs, alloy 5182 was treated



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according to the process of Patent 3,839,019 and ingots thereof were cast. In further runs, the same procedure was employed except that the process of Patent 3,839,019 was followed by the improved process as depicted in Figure 2, including the use of a chlorine-argon gas mixture introduced into zone 140. The improved system included a bed/38 of Raschig rings 1/2 inch outside diameter, 3/8 inch inside diameter and 1/2 inch long, the bed being about 13 inches deep in main flow path (bottom of baffle 118 to top of bed). Five inclined channels were provided between parallel surfaces in zone 136. The results are shown in Table 2 below.

Table 2

	Na		Ca		Oxide Patches
	Before	After	Before	After	
Patent 3,839,019 Only	.004	.0003	.003	.0004	Serious oxide patches
Patent 3,839,019 plus improvement	.004	.0000	.003	.0002	No oxide patches

It can be seen that the present improvement eliminates the ingot oxide patches completely but, further, facilitates still more removal of Na and Ca than the process of Patent 3,839,019 along, it being noted that said Patent 3,839,019 process has enjoyed considerable commercial use because of its recognized benefits. Yet, the use of said process is greatly enhanced by the present improvement which not only greatly improves removal of Na and Ca but completely eliminates salt-derived oxide patches from ingot surfaces sometimes encountered with aluminum alloys containing substantial amounts of magnesium.

Tests on molten aluminum have further verified the effectiveness of the present improvement, especially in removing salt containing impurities. Melt samples were taken at points corresponding in Figure 2 to the inlet portion 120 (before treatment), zone 148 (intermediate) and outlet bay 130 (after treatment). The samples were



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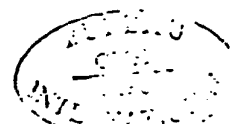
- solidified in graphite sample containers which were sectioned for examination at 50x magnification. Salt impurities absorb moisture so rapidly when exposed to the atmosphere that as the metal solidifies the salt and moisture form an enlarged spherical "globule". Such spherical globules provide both qualitative and quantitative indication as to salt impurities present. Examination of the sample for the inlet (untreated) metal revealed a plurality of spherical globules, several spheres of different sizes being visible.
- 10 Examination of aluminum from zone 148 exhibited greatly reduced frequency (less than half) and significantly reduced size of globules. Examination of the aluminum in outlet bay 130 indicates no globules present, thus further verifying the effectiveness of the improved system.
- 15 While the foregoing description has emphasized the improvement as applied to treating molten aluminum, such is not necessarily intended to limit the scope of the improvement herein described which applies to the light metal magnesium and to other metals. While considerable emphasis
- 20 has been placed on buoyant salt-bearing particles, the invention has demonstrated the capability of removing nonbuoyant particles such as titanium diboride (TiB_2) particles and aluminum and other oxide impurity particles.
- 25 While the invention has been described in terms of preferred embodiments, the claims appended hereto are intended to encompass other embodiments which fall within the spirit of the invention.



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C L A I M S

1. A method of treating molten metal containing suspended particles to remove said particles from said metal comprising passing said metal through a medium of submerged noncontaminating contacting surfaces, said medium having an average void fraction of at least one-half and a specific surface area of at least 25 sq. ft. per cubic ft.
2. The method according to claim 1, wherein said specific surface area is 75 square feet or more.
3. The method according to claim 1 or 2, wherein said void fraction is 0.7 or more.
4. The method according to claim 1 or 3, wherein said specific surface area is 50 square feet or more.
5. A method according to claim 1, wherein said metal moves through said medium at a superficial metal velocity of not more than one-half ft./minute.
6. The method according to claim 5, wherein said metal moves through said medium at a superficial metal velocity of 0.4 ft./minute or less.
7. The method according to any one of the preceding claims, wherein said molten metal moving through said submerged noncontaminating contacting surfaces is contacted with a gas.
8. The method according to claim 7, wherein said gas moves in counterflow relationship with said molten metal.
9. The method according to claim 7, wherein said gas moves in concurrent flow relationship with said molten metal.
10. The method according to any one of the preceding claims, wherein periodically (and not continuously) a gas is passed through said medium at a sufficient rate to disturb said medium sufficiently to dislodge particles previously removed from molten metal passing through said medium and remove said particles from said medium, said particles rising upwardly from said medium (by the action of said gas).
11. The method according to claim 10, wherein molten metal movement through said medium is interrupted during said



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gas purge of said medium.

12. The method according to any one of the preceding claims, wherein upward media movement is restrained by means situated upon said media.

13. A method according to any one of claims 1 to 3, wherein said suspended particles include agglomerable fine suspended particles which are treated to agglomerate said particles to a size separable by gravity from said molten metal and facilitate removal of said particles from said molten metal, by passing said metal containing said particles through said medium of submerged noncontaminating contacting surfaces so as to agglomerate portions of said particles and render them separable by gravity from said molten metal.

14. The metal according to claim 13, wherein the average superficial velocity of said molten metal moving through said medium of submerged noncontaminating surfaces is not more than five feet per minute.

15. The method according to claim 13 or 14, wherein the average superficial velocity of said molten metal moving through said medium of submerged noncontaminating surfaces is greater than one-half foot per minute.

16. The method according to claim 15, wherein said superficial velocity is 0.6 foot per minute or more.

17. The method according to any one of claims 13 to 15, wherein the average superficial velocity of said molten metal moving through said medium of submerged noncontaminating surfaces is from one-half to three feet per minute.

18. The method according to any one of claims 13 to 17, wherein said molten metal containing particles agglomerated in said medium of submerged noncontaminating contacting surfaces is moved through at least one inclined channel bounded by at least one inclined surface, said movement through said channel being laminar in flow to remove said particles by gravity from said moving molten metal to said inclined surface.

19. The method according to claim 18, wherein a plurality of inclined channels provide pluralities of said



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inclined surfaces.

20. The method according to claim 18, where in a plurality of inclined channels are provided between substantially parallel surfaces.
21. The method according to claim 18, wherein a plurality of inclined channels are provided between substantially flat parallel surfaces.
22. The method according to claim 21, wherein said surfaces are approximately one-half to two inches apart.
23. The method according to claim 18, wherein a plurality of channels are provided by a plurality of inclined substantially parallel planes.
24. The method according to claim 18, wherein a plurality of channels are provided by a plurality of inclined substantially parallel tubes.
25. The method according to claim 20, wherein said surfaces are inclined at an angle between 10° and 70° from the horizontal.
26. The method according to claim 20, wherein said surfaces are one-half to two inches apart and are inclined at an angle between 20° and 45° from the horizontal.
27. The method according to any one of claims 18 to 26, wherein said metal moves downwardly and laterally through a plurality of inclined channels and agglomerated particles collect on the upper surfaces bounding said channels and move upwardly and rise off the upper portions of said upper surfaces.
28. The method according to any one of claims 18 to 27, wherein said molten metal is molten aluminum containing suspended particles in the range of one to 50 microns which are agglomerated in said media of submerged noncontaminating contacting surfaces into particles above 50 microns which are separable by gravity from moving molten aluminum.
29. The method according to any one of claims 18 to 28, wherein molten metal movement through said inclined channel is characterized by a Reynolds number not exceeding 3500.
30. The method according to claim 29, wherein molten



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metal movement through said inclined channel is characterized by a Reynolds number not exceeding 2800 and by substantial freedom from turbulence or flow separation from said inclined surface.

31. The method according to any one of claims 18 to 27, wherein said molten metal is a light metal selected from aluminum and magnesium.

32. The method according to any one of claims 18 to 31, wherein said molten metal is aluminum.

33. The method according to any one of claims 18 to 32, wherein said particles comprise buoyant halide salt.

34. The method according to any one of claims 18 to 33, wherein said particles comprise liquid buoyant halide salt.

35. The method according to any one of claims 18 to 34, wherein a gas is introduced into said medium of noncontaminating contacting surfaces.

36. The method according to claim 35, wherein said gas comprises a nonreactive gas.

37. The method according to claim 35, wherein said gas comprises a halogenaceous gas.

38. The method according to claim 35, wherein said gas comprises a mixture of a nonreactive gas and a halogenaceous gas present in amounts less than said nonreactive gas.

39. The method according to any one of claims 18 to 38, wherein a gas is introduced into the medium of submerged noncontaminating contacting surfaces at a first gas flow rate and wherein said gas flow rate is periodically increased to purge and dislodge agglomerated particles from said media and cause said agglomerated particles to rise from said media to form a phase substantially floating upon said molten metal.

40. The method according to claim 39, wherein said increased gas flow rate is at least two times said first gas flow rate.

41. The method according to claim 39 or 40, wherein molten metal movement through said submerged contacting surfaces media is interrupted during said period of increased gas flow rate.



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42. The method according to any one of claims 18 to 41, wherein said molten metal is passed in downward course through said medium of noncontaminating contacting surfaces.

43. The method according to any one of claims 18 to 42, wherein the average retention time of said molten metal in said medium of submerged contacting surfaces is three-fourths minute or longer.

44. The method according to any one of claims 18 to 43, wherein said molten metal is moved downwardly through a first medium of said submerged noncontaminating surfaces and then upwardly through a second medium of said submerged noncontaminating surfaces.

45. The method according to claim 44, wherein said metal moves countercurrent to rising gas in said first medium and moves concurrent with rising gas in said second medium.

46. The method according to any one of the preceding claims, wherein said noncontaminating surfaces are refractory (alumina for aluminum).

47. The method according to any one of the preceding claims, wherein said submerged noncontaminating surfaces in said medium comprise ring-shaped bodies.

48. The method according to any one of the preceding claims, wherein said submerged noncontaminating surfaces in said medium comprise saddle-shaped bodies.



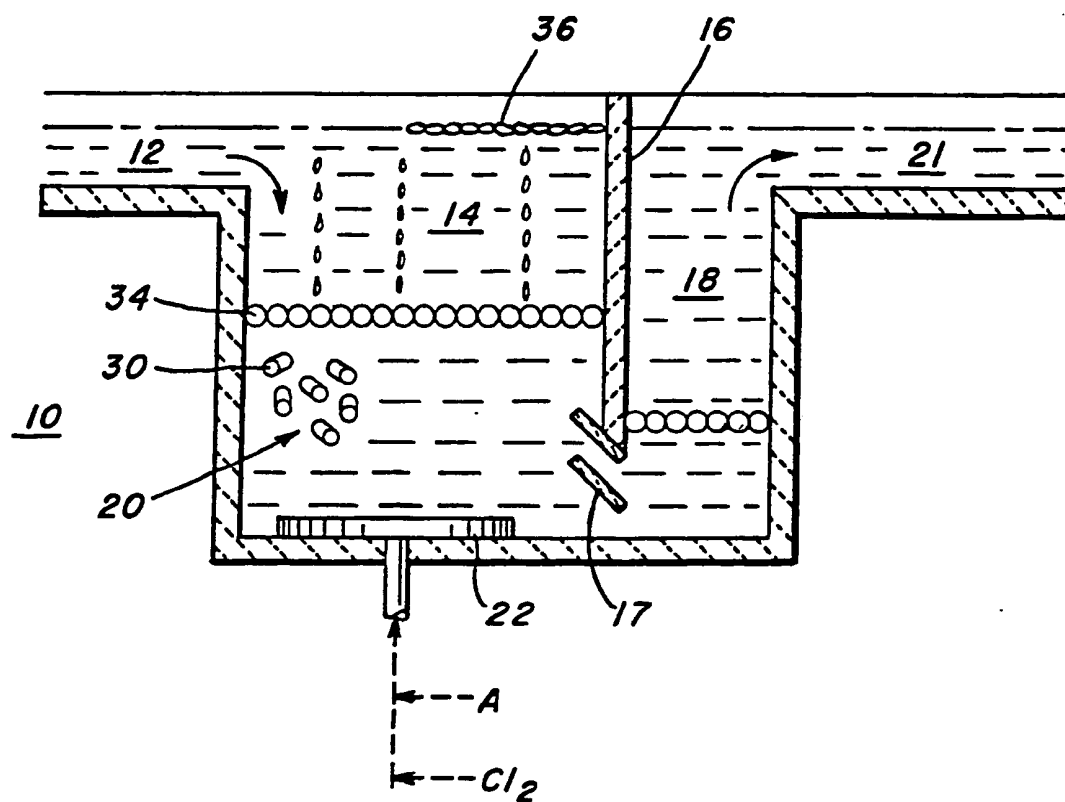
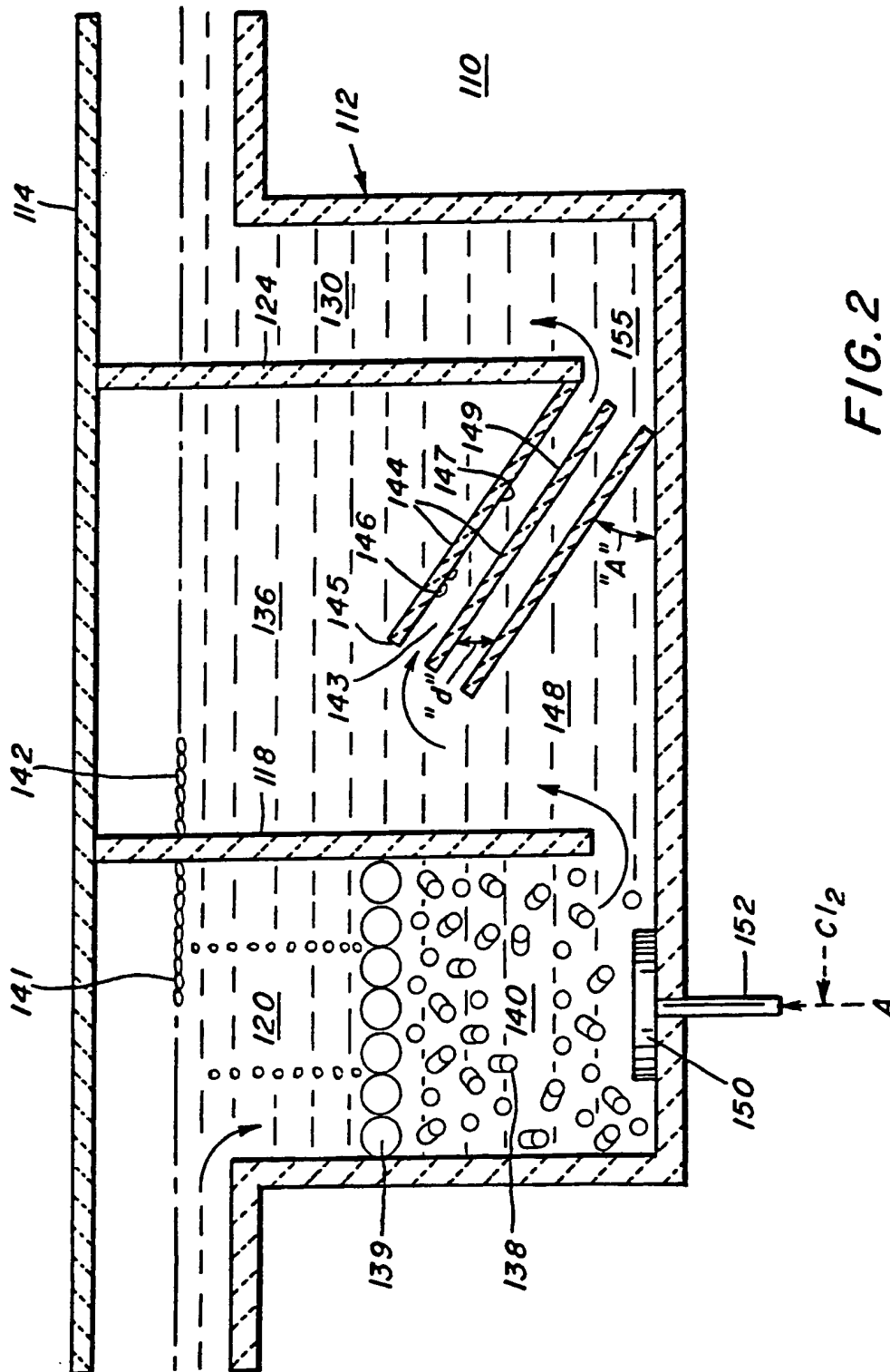


FIG. 1



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SUBSTITUTE SHEET



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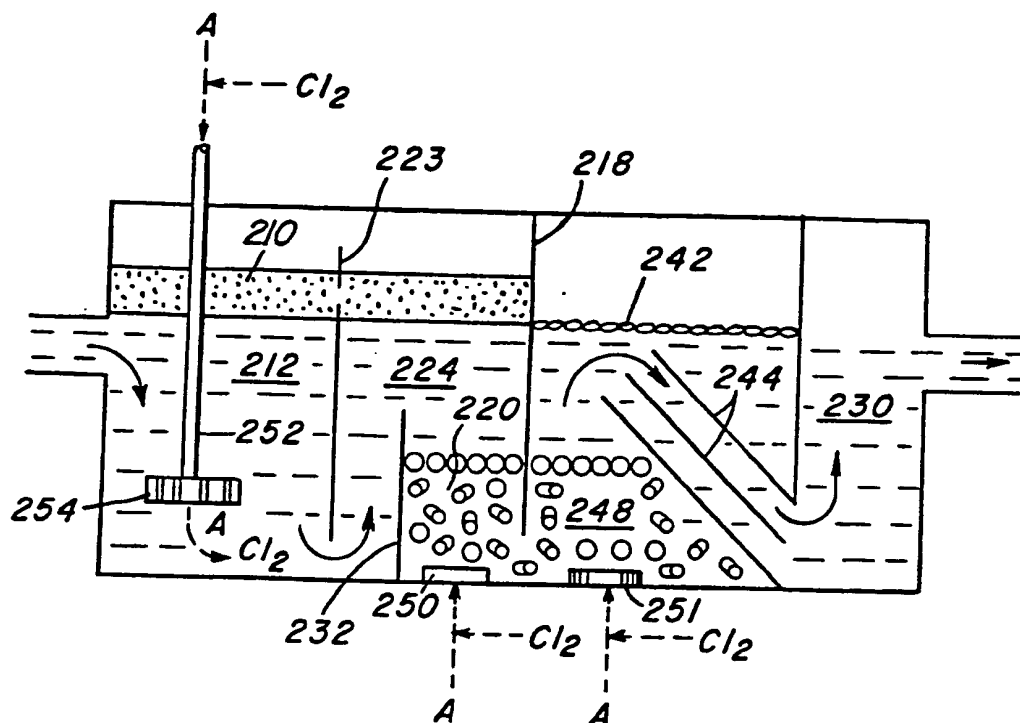


FIG. 3

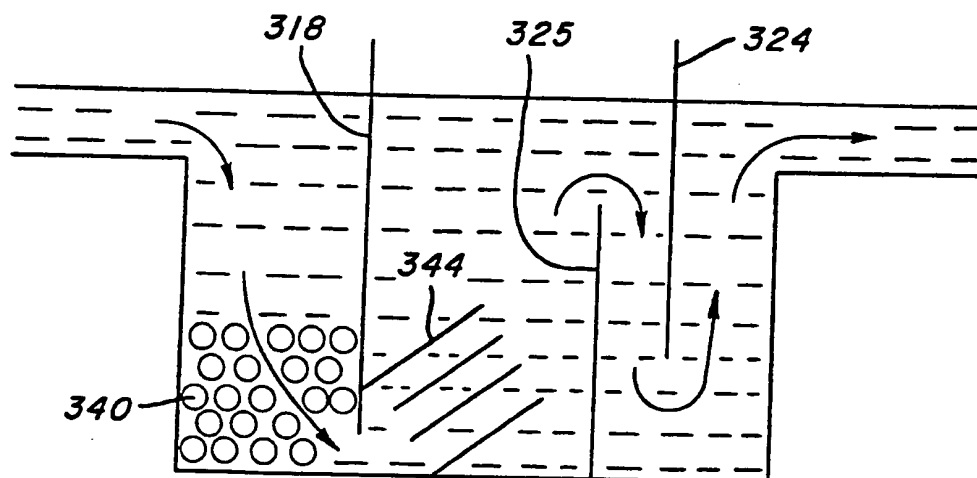


FIG. 4

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